

**LABORATORY SUPPORT FOR in situ
GASIFICATION REACTION KINETICS**

**Quarterly Report for the Period
October—December 1976**

by

**J. Fischer, J. E. Young,
J. E. Johnson, and A. A. Jonke**

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LABORATORY SUPPORT FOR *IN SITU* GASIFICATION REACTION KINETICS

by

Jack Fischer, J. E. Young, J. E. Johnson, and A. A. Jonke

ABSTRACT

This work, which is part of the ANL energy program for ERDA, is directed toward support studies for the national endeavor on *in situ* coal gasification. The objective of this work is to determine the reaction-controlling variables and reaction kinetics for gasification of chars resulting when coal is pyrolyzed in underground gasification. The reactions to be studied include steam-char, CO₂-char, H₂-char, and the water gas shift reaction.

Discussed in this report are the effects on the reactivity of chars of pyrolysis in simulated gasification product gas rather than pure nitrogen. In addition, further investigations of the water gas shift reaction and methanation reaction are discussed.

SUMMARY

This work has the objectives of providing engineering data for process control and resource evaluation for underground coal gasification. This is to be accomplished by providing data for gasification of chars under simulated *in situ* processing conditions. The coal samples studied are representative of coals at sites where field tests either are currently being conducted or are under consideration. Chars for gasification are prepared by pyrolyzing the coal under conditions characteristic of the *in situ* gasification processes. Studies carried out during this quarter indicate that pyrolysis of coal in simulated product gases (reducing atmosphere) has little effect on the reactivity of the resultant char compared to the reactivity of char resulting from pyrolysis of coal in nitrogen.

Other studies carried out this quarter suggest that although the metals and alumina packing in our reactor system contribute to the catalysis of the water gas shift reaction, the primary catalytic agent is the mineral matter in the coal char, and that thermodynamic equilibrium is reached for this reaction at temperatures as low as 600°C for a gas residence time of 0.1 min.

We have also found that the methanation reaction and the carbon monoxide disproportionation reaction are catalyzed by the reactor and packing materials but that further studies are necessary to determine the role of coal minerals in these reactions.

PROJECT PLAN AND STATUS

The project plan is shown in Table 1. Studies are currently being carried out to improve the reliability of kinetic parameters previously determined for the reaction of steam with chars from Wyodak and Hanna coals. It is expected that these studies will be finished by the proposed completion date of April 1, 1977, for this portion of the project. The steam-char kinetics for Pittsburgh seam coal, kinetics of the CO_2 reaction with western and eastern char, the water gas shift reaction and effects of brackish water constituents on kinetics of char gasification will be completed in the remainder of FY-1977, and a final report will be written in September 1977.

CONCLUSIONS

1. Pyrolysis of coals in a reducing atmosphere results in chars which have a reactivity with steam essentially equal to that for chars pyrolyzed in nitrogen. Although previous studies have indicated that pyrolysis in reducing atmospheres results in a greater fraction of coal being volatilized, our studies indicate that residual chars prepared in the two atmospheres have the same reactivity with steam. This finding confirms that results of earlier experiments (carried out as a part of this project) involving the preparation of the chars in nitrogen are relevant to mathematical models for underground gasification of coal.

2. The water gas shift reaction is very effectively catalyzed by the mineral matter in subbituminous coal chars. At temperatures as low as 600°C , thermodynamic equilibrium is reached in a gas residence time of 0.1 min. Our metal reactor system and alumina packing materials contribute somewhat to the catalysis of this reaction, but especially at lower temperatures (e.g., 600°C), this effect is insignificant.

3. The methanation of CO by hydrogen and the disproportionation of CO to carbon and CO_2 both occur throughout the temperature range of $500\text{--}800^\circ\text{C}$. These reactions are catalyzed by the surface of the high-purity α -alumina used in our reactor system and probably by the mineral matter present in the coal chars.

INTRODUCTION

Current and predicted future shortages of energy in this country have led to increased effort to develop processes for the underground conversion of coal to cleaner fuels or petrochemical feedstocks. The concept of *in situ* coal gasification has been investigated intermittently in the U.S.A. and European countries since the early part of this century. Extensive testing of the concept has been carried out in the U.S.S.R. However, underground gasification development has been interrupted in the past either by war or by alleviation of the need for gasification as a result of the discovery of extensive reserves of natural gas (as in the case of the Soviet Union). The increased interest in underground gasification in this country can be attributed to the shortage of gaseous fuels and to a number of potential advantages:

Table 1. Laboratory Support for *In Situ* Gasification
Reaction Kinetics

[illegible]

1. Recent cost evaluations indicate that capital expenditure for underground gasification would be lower than the combined cost of strip or deep shaft mining and aboveground conversion to comparable gaseous fuels by Lurgi gasifiers.
2. Less disruption of the environment is caused by *in situ* gasification than by aboveground processing.
3. Coal that cannot be economically mined and utilized aboveground could be gasified underground.
4. Personnel would not be subjected to hazards associated with underground mining.
5. Gas produced *in situ* can be used for local power production or as chemical feedstock, or can be converted to synthetic pipeline gas.

During gasification of coal underground, three distinct reaction zones can be identified. In the first zone, drying and pyrolysis (devolatilization) of the coal occur. In a second zone, a portion of the char is gasified by reaction with steam and carbon dioxide. Combustion of the remaining char by air or oxygen injected into the coal seam occurs in the third zone where carbon dioxide is produced. The combustion zone generates the heat for the process, which is carried into the other zones by the flow of the hot gaseous products of combustion.

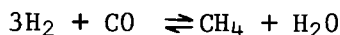
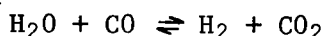
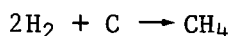
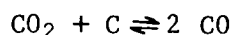
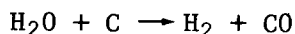
Mathematical models are currently being developed at other laboratories in order to describe with mathematical formulas the time and spatial dependence of the gasification processes as they occur underground. These models are necessary if *in situ* gasification is to be employed in an efficient and economic manner to produce a variety of products, utilizing a variety of coal sources. Petrochemical feedstocks, combined-cycle power generation, and pipeline gas production will require product gases of different compositions; each application could make good use of products of *in situ* gasification. An effective mathematical model can be used to determine how variables such as air injection rate, water infusion, and steam partial pressure can be adjusted to tailor the product to the needs of the consumer.

In addition to prediction of product gas compositions, models permit calculation of the efficiencies of the process, prediction of rates of movement of the various reaction zones during *in situ* gasification, specification of optimum process configurations and parameters, and prediction of the effects of processing upsets, e.g., massive subsidence into the reaction zone. Not all of those capabilities have yet been included in current models for *in situ* gasification, but refinements will be made to permit use of the models to meet these as well as other goals.

Kinetic information used in current mathematical models have been obtained from studies with coals and operating conditions unlike those encountered in underground gasification. The purpose of our work at ANL is to obtain kinetic information that is directly applicable to mathematical models for *in situ* gasification. These studies will allow the important processing variables in underground gasification to be identified. The coals utilized are similar to

those used in current field tests or proposed for use in commercial underground gasification. The processing conditions surveyed include those expected to be encountered in *in situ* gasification.

The kinetics of the reaction of oxygen with carbon are not included in our work. This reaction is rapid, and the results obtained in the mathematical models are quite insensitive to errors in the rate constants for this reaction. The reactions being studied in this program include:



Only the first three of the above reactions contribute directly to conversion of char to gaseous products. The final two reactions affect the composition of the product gases and are of considerable economic importance in relation to tailoring the product gas to the needs of various industries which would make use of the end products of *in situ* gasification.

Variables being investigated in this study include total pressure, reaction temperature, coal devolatilization conditions, and partial pressure of steam. Kinetic data to be obtained include rate constants, reaction orders with respect to each of the reactants, and apparent activation energies. These parameters are to be determined for bituminous coal from the Pittsburgh seam and are being determined for subbituminous coals from Wyodak and Hanna seams of Wyoming. In addition, the effects on the reaction kinetics of introducing various constituents of brackish water will be determined.

In this quarterly report are described the results of a number of experimental runs carried out to investigate: (1) the effects of gasification rates of pyrolyzing the coal in a simulated gasification product mixture rather than in pure nitrogen, (2) the water gas shift reaction as catalyzed by mineral matter naturally occurring in the coal, and (3) the methanation reaction ($\text{CO} + 3\text{H}_2 \rightleftharpoons \text{CH}_4 + \text{H}_2\text{O}$) as catalyzed by the coal mineral matter.

RESULTS AND DISCUSSION

The experimental apparatus and operating procedures have been described in detail in ANL-76-131.

Pyrolysis in Simulated Product Gas

As pointed out in ANL-76-131, it is important that in laboratory work pyrolysis of coal prior to gasification be carried out under conditions closely simulating those encountered underground. We have shown that a result of

temperature cycling and/or exposure to air between the pyrolysis and gasification steps is a char that is less reactive with steam than is a char pyrolyzed immediately prior to gasification. Since one would not anticipate that underground processing would involve temperature cycling or air exposure prior to gasification under normal circumstances, all of our studies are being carried out with the time between the pyrolysis and gasification steps minimized. The heating rate used during our pyrolyses is approximately that expected in underground processing, i.e., $3^{\circ}\text{C}/\text{min}$.

In previous experiments, we have used nitrogen to sweep the pyrolysis products from the coal bed. In underground gasification, this sweeping gas would actually be the product gas from the gasification reaction zone, i.e., the gas would contain hydrogen, carbon monoxide, carbon dioxide, small amounts of methane, and nitrogen. Previous studies carried out at Lawrence Livermore Laboratories¹ indicated that varying the composition of the gas present during pyrolysis of subbituminous coal results in a change in the amount of volatile matter pyrolyzed from the coal. However, the effects of these variations in pyrolysis sweeping gas on the reactivity of the remaining char have not been determined previously.

Two gasification experiments have been carried out using experimental conditions that were similar except for the pyrolysis sweeping gas. In one test, nitrogen was used during pyrolysis; in the other test, a mixture consisting of 20% H_2 , 20% CO , 10% CO_2 , and 50% N_2 was used during pyrolysis. Wyodak subbituminous coal was pyrolyzed at a heating rate of $3^{\circ}\text{C}/\text{min}$. The total gas pressure was 1.60 MPa (16.3 atm). Final pyrolysis temperature and the gasification temperature were both 700°C . Steam partial pressure during gasification was 0.73 MPa (7.4 atm).

Figure 1 shows the reaction rate for the steam/char reaction for coal that has been pyrolyzed in nitrogen. The reaction rate for coal pyrolyzed in the simulated product gas (reducing gas) is shown in Fig. 2. The reaction rate is expressed as grams of carbon gasified per hour per gram of ash-free carbon present, and is plotted as a function of the percent of the char present in the reactor which has been converted to gas. Comparison of Figs. 1 and 2 shows little if any effect on the reactivity of the Wyodak char caused by the difference in pyrolysis sweeping gas. The maximum reaction rates are nearly the same in both cases, and the general shapes of the curves are similar.

In the balance of our kinetics studies, we will carry out the pyrolysis step using a reducing gas mixture as the sweeping gas. However, the information obtained in earlier experiments utilizing nitrogen as the sweeping gas is still valid and applicable to mathematical models for underground gasification.

Water Gas Shift Reaction

As described in ANL-76-131, the distribution of product gas constituents indicates that the water gas shift reaction is very nearly at thermodynamic equilibrium for our reaction conditions and is most likely catalyzed by the mineral matter occurring in the coal. The ratio of H_2 to CO_2 is generally nearly 2:1, and the concentration of CO in our product gas is usually nil. Throughout the temperature range of our studies at this time ($600\text{--}750^{\circ}\text{C}$), the carbon monoxide concentration in our product gas has been below the detection limit of our chromatograph.

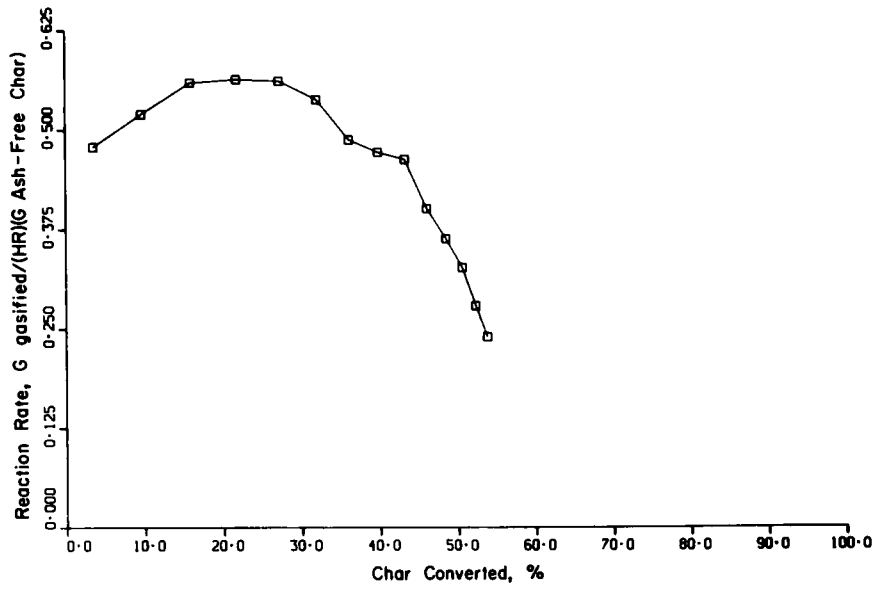


Fig. 1. Instantaneous Carbon Conversion Rate vs. Percent Wyodak Char Converted after N_2 Pyrolysis

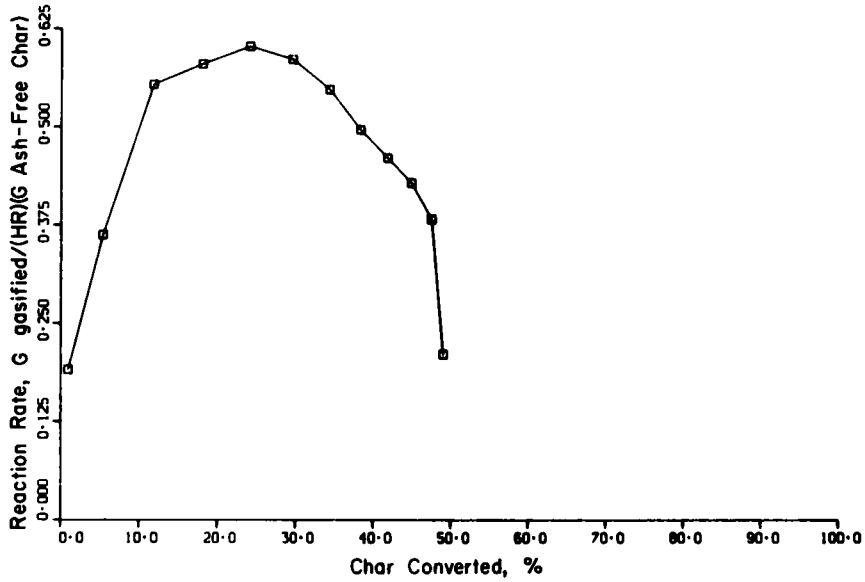


Fig. 2. Instantaneous Carbon Conversion Rate vs. Percent Wyodak Char Converted after Reducing Pyrolysis

Since these gasification kinetics studies are carried out in a differential reactor, the coal or char bed fills only a very small fraction of the volume of the reactor. The balance of the reactor is filled with high-purity alumina. Although this inert material contains no impurities which would be expected to catalyze the water gas shift reaction, a large surface area is available which might promote the heterogeneous reaction of steam with CO. For this reason, blank runs have been made to determine the extent of catalysis of the water gas shift reaction by the reactor material and inert packing materials usually used in the kinetics experiments. The reactor consists of 316 stainless steel pipe, the interior of which has been electroplated with a 0.025 mm layer of gold. The packing material used in the reactor is Norton RR high-purity alundum. A blend of 2% CO in nitrogen was passed through the reactor at a pressure of 1.60 MPa (16.4 atm). Steam was introduced at a partial pressure of 0.51 MPa (5.3 atm). Temperature was varied from 600 to 800°C. The residence time of the gas mixture in the reactor was 0.3 min.

A reaction constant can be calculated according to the relationship:

$$k_p = \frac{[P_{CO_2}] [P_{H_2}]}{[P_{H_2O}] [P_{CO}]}$$

where P_{CO_2} = partial pressure of CO₂

P_{H_2} = partial pressure of H₂

P_{H_2O} = partial pressure of steam

P_{CO} = partial pressure of CO

The partial pressure of each component at the outlet of the reactor was used in this calculation. The calculated reaction constants for the reactor with alumina packing and for the empty reactor are compared in Fig. 3 with equilibrium constants calculated from thermodynamic data. Even at 750°C, the reaction constant for the empty gold-plated reactor was three orders of magnitude smaller than that calculated from thermodynamic data. However, for a contact time of 0.3 min, the alumina packing in the reactor has sufficient catalytic activity that the water gas shift reaction reaches thermodynamic equilibrium at 800°C.

In the temperature range of interest for the steam/char reaction studies (550–800°C), the contribution of the reactor and alumina packing to the catalysis of the water gas shift reaction is insignificant. Hence, catalysis of this reaction that was observed in previous runs at temperatures as low as 600°C can be attributed only to the char bed itself--most likely to the mineral matter in the char.

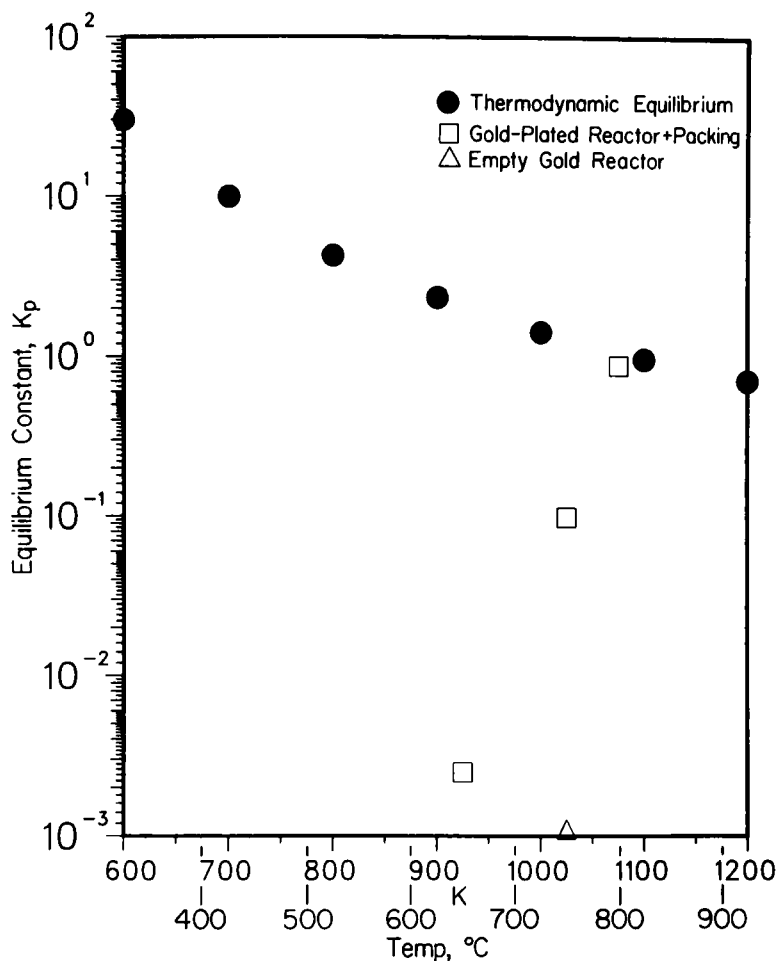


Fig. 3. Water Gas Shift Reaction Equilibria

Methanation Reactions

In ANL 76-131, the observation was made that the presence of steam promotes the reaction of hydrogen and char, resulting in more methane being produced than was observed in the absence of steam. This phenomenon could be the result of either direct interaction of carbon and steam or by occurrence of the methanation reaction



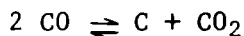
catalyzed by the mineral matter present in the char. Goring *et al.*² have postulated the former model but could not describe in detail a mechanism for this direct interaction of steam and carbon to produce methane.

We have carried out a number of experiments in an attempt to clarify this question. Mineral matter obtained by low-temperature ashing of Hanna seam subbituminous coal was placed in our gasification reactor, supported on a bed of high purity α -alumina. A blend of approximately 10% CO and 10% H₂ in nitrogen was passed over the bed of ash. The total pressure was 1.60 MPa (16.4 atm). Hence the partial pressures of hydrogen and carbon monoxide were each approximately 0.16 MPa (22 psig).

An equimolar concentration of hydrogen and carbon monoxide is not the optimum stoichiometric ratio for the methanation reaction. However, it is undoubtedly close to the concentration ratio expected in the steam-char gasification zone of the process as it occurs underground.

The average residence time of the gas mixture in the reactor was 0.3 min. Throughout the temperature range of 550 to 800°C the measured methane concentration at the reactor outlet was approximately two orders of magnitude lower than that expected if the reaction had reached thermodynamic equilibrium in this residence time. However, under our operating conditions (differential conversion) this corresponds to rather high conversion of CO to CH₄.

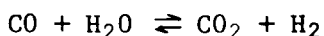
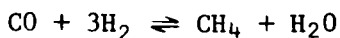
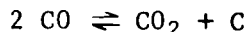
However when the reactor was dismantled, large amounts of soot were found in the alumina packing above the coal ash bed. The presence of this soot indicates the occurrence of the competing disproportionation reaction:



This would result in a considerably lower concentration of carbon monoxide at the location of the coal ash in the reactor.

A repeat of this experiment with fresh alumina packing and no coal ash present in the reactor again yielded a relatively high methane content in the outlet gas and a soot bed was again generated at the entrance to the reactor hot zone. In fact, yet another repeat of this experiment in the empty gold-plated reactor gave relatively high methane yields. Within a short time, this experiment had to be terminated because of excessive pressure drop across the reactor due to choking of the reactor by soot that had collected in the reactor outlet.

As a consequence, no firm conclusions can be made regarding the mechanism of methane production by the reaction of steam and hydrogen with char. The reactions of carbon monoxide in the char bed are apparently quite complex, with the following reactions all occurring to an appreciable extent:



Considerable more investigation of this question is necessary in order to obtain information that will be useful in mathematical models for the underground gasification process.

FUTURE WORK

During the next quarter, we expect to complete the determination of the kinetic parameters for the reaction of steam with chars prepared from Wyodak and Hanna subbituminous coals. We also expect to begin investigation of the reaction of steam with chars prepared from Pittsburgh seam coal. Further investigation of the water gas shift and methanation reactions will be postponed until later quarters.

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